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A SMALL-SCALE TEST FOR FIBER RELEASE FROM CARBON COMPOSITES*

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SUMMARY

The release of electrically conductive fibers from resin/carbon fiber composites presents a potential problem to electronic equipment. A test method was developed to determine fiber loss from pyrolyzed composites. Eleven composites consisting of woven and unwoven carbon fiber reinforcement and different resins were subjected to the burn and impact test device. The composites made with unidirectional tape had higher fiber loss than those with woven fabric. Also, the fiber loss was inversely proportional to the char yield of the resin. This technique provides a method to determine relative fiber loss with different resins and fiber construction.

INTRODUCTION

Composites consisting of carbon fibers and a matrix resin have been used in a wide variety of applications from sports equipment to aircraft parts. The high strength, stiffness, and low weight of carbon composites provide sufficient advances so that it is reasonable to assume that greater use of this material will be seen in the future.

Early in 1978 it was reported (ref. 1) that the release of free carbon or graphite fibers into the environment was a potential problem. The high electrical conductivity of the fibers and the ease of mobility of the free fibers present problems when they settle across electrical contacts. This could result in failure of equipment. An accidental fire on an aircraft could result in significant numbers of free fibers of carbon being released and, if the fibers reach the electronics, a serious malfunction could occur.

We have been studying fire-resistant resins for aircraft applications for a number of years (ref. 2). It has been reported (ref. 3) that the higher the char yield the more resistant the resins are to ignition and burning. Therefore, if these high-char-yielding resins are used as the resin matrix in carbon composites, less fibers would be released into the atmosphere.

To evaluate the fiber release from composite materials, we have designed a new burn/impact test apparatus as a screening device to determine the amount of fiber release after burn and impact. We have also calculated the theoretical char binder content of the composites when exposed to the burner.

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DISCUSSION

The equipment used to evaluate the burn/impact properties of carbon composites is shown in figure 1. The test specimen consists of carbon composites with various resin matrices which were

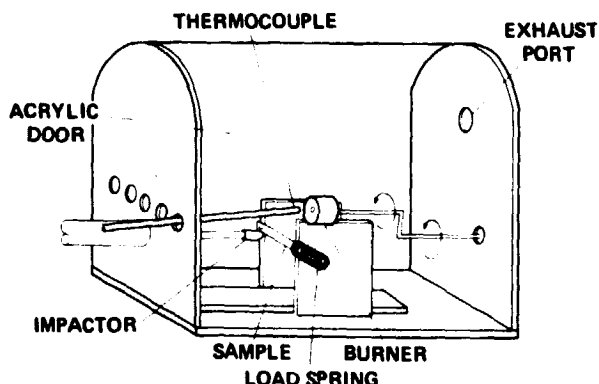


Figure 1.— Ames burn-impact unit.

machined to dimensions of 133 mm (5.25 in.) long and 12.7 mm (0.5 in.) wide and a thickness as received (2.3 to 3.5 mm) but preferably 3.1 mm (0.125 in.). The test specimens were weighed and mounted in the holder and a tensile load of 444.8 N (100 lb) was applied to the test specimen by compressing the spring. The burner was supplied with a mixture of air (13.1 liters/min) and propane gas (0.55 liters/min) and ignited. These values for air and propane are equivalent to 5 mol of oxygen per mol of propane which would make a nonoxidizing flame. The burner was allowed to heat up for 3 min and the temperature in the warmup position with the

door open was 927°-100° C (1700°-1850° F). A shop vacuum was attached to the exhaust port which was covered with "bridal veil" net coated with a tacky polyvinyl chloride adhesive (Rhoplex N619). The burner was rotated into position and the sample was burned for 3 min. The burner was then shut off and the specimen was allowed to cool 5 min. The specimen was then weighed and placed back in the fixture and the load of 444.8 N was applied. The specimen was then impacted at an impact velocity of 12.2 m/sec with the piston from the air cylinder. The load on the air cylinder was 0.648 MPa (94 psi). After the specimen was impacted it was removed and weighed.

The percent of fiber weight loss from impact was calculated as:

$$\% \text{ Impact loss} = \frac{\text{Weight after burn} - \text{weight after Impact}}{\text{Original weight (60\%)}} \times 100$$

The calculation of the char binder of the composite was made based on the temperature of the test specimen and the char yield of the resin as determined by thermogravimetric analysis. In the burn-impact procedure, the 50-mm (2-in.) diam heater was mounted at a distance of 20.0 mm (0.8125 in.) from the test specimen. Because of conductive heat flow through the test specimen, there is a resulting thermal profile across the specimen (see fig. 2). From the center to a point 12.7 mm (0.5 in.) on either side of the centerline, the maximum temperature was 750° C (1385° F). Nineteen mm (0.75 in.) on either side of the centerline, the maximum temperature was 649° C (1200° F). At a distance of 25.4 mm (1.0 in.) on either side of the centerline, the temperature was 520° C (968° F). At 38.1 mm (1.5 in.) on either side of the centerline, the maximum temperature was 427° C (800° F). Thus, if the temperature gradient, resin content (% wt), and char yield are known, the percent char binder can be calculated.

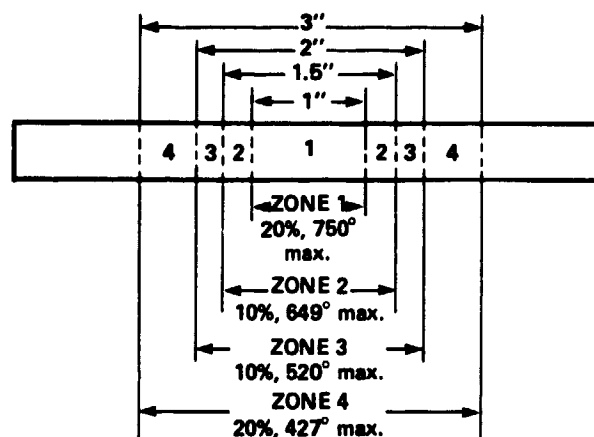


Figure 2.— Thermal profile of burn test specimen.

The theoretical char binder of the composite is calculated as:

$$\text{Calculated char binder} = \frac{R'}{\text{Specimen weight} \times 60\%}$$

where:

$$R' = R(0.2)(Y_{c750^{\circ}\text{C}}) + R(0.1)(Y_{c649^{\circ}\text{C}}) + R(0.1)(Y_{c520^{\circ}\text{C}}) + R(0.2)(Y_{c427^{\circ}\text{C}})$$

$$R = \text{Specimen weight} \times \text{resin content (\% weight)}$$

In this equation the weight fraction of each temperature zone is multiplied by the char yield (table 1). The sum of the char yield for each zone is divided by 60% of the weight of the specimen to give the percent char binder. The original specimen weight is normalized to 60% because this is the portion of the specimen that is heated to a temperature above 280° C (536° F). Below that temperature, the weight loss is insignificant (5% or less).

An example of the calculated char binder for an epoxy/carbon composite with a resin content of 33.2% (wt) and the char yield at 750° C of 36%, at 649° C of 37%, at 520° C of 41%, and at 427° C of 56%, with a sample weight of 8.02 g, is 14.5%.

TABLE 1.— RESIN CHAR YIELD — THERMOGRAVIMETRIC ANALYSIS

RESIN TYPE	PERCENT CHAR YIELD			
	750° C	649° C	520° C	427° C
POLYSTYRYL PYRIDINE	68	72	80	96
EPOXY	36	37	41	56
PHENOLIC - A	58	60	72	84
PHENOLIC - B	54	55	68	92
PHENOLIC - C	61	64	76	88
POLYIMIDE	55	58	72	86

TEST RESULTS

Eleven test composites were prepared using the processing conditions given in table 2. Five specimens of each were subjected to the burn/impact procedure. The resin content, resin type, fiber type, calculated char binder, and percent weight loss after burn and impact are summarized in table 3. The resins used included epoxy, phenolic, polystyryl pyridine, and polyimide.

The carbon reinforcements used include unidirectional tape, satin weave, and plain weave. The samples listed in table 3 are grouped according to the type of reinforcement. The first two samples were made with unidirectional tape in a "quasi-isotropic" layup (0, ± 45 , 90); the next seven samples were made with a satin weave (T-300, 24X24 yarn count, 11 oz/yd²) cloth; and the last two samples were made with plain weave (T-300, 12X12 yarn count, 5.5 oz/yd²) cloth. The quasi-isotropic samples had higher fiber loss (as determined by weight loss after burn and impact) than the woven samples. For example, the epoxy resin quasi-isotropic composite had fiber release of 12.6% with calculated char binder of 13.1%; the same resin on satin weave cloth had fiber release of 1.02% with char binder (cal.) of 14.5%; on a plain weave cloth sample, the fiber release was 0.9% with calculated char binder of 11.4%.

Because the majority of the samples tested was made with satin weave cloth, a comparison can be made between the different resin systems and in two cases comparing two different resin contents with the same resin type. The test results listed in table 3 are ranked according to the calculated char binder. The values for the calculated char binder are listed with the highest values at the top. The values for percent impact loss and calculated char binder are also presented in figure 3. It can be seen that the higher the char yield the lower the impact loss. Samples with the same type of resin (phenolic-2), such as S-2 and S-4, show that with higher resin content there is necessarily a higher char binder and less fiber release after burn and impact. The same results held true for the two epoxy samples (S-6 and S-7). The upper values for achieving improved fiber retention is not known, but within the resin contents employed there is a correlation between resin content and fiber release.

TABLE 2.— COMPOSITE PROCESSING

RESIN (SAMPLE NUMBER)	CURE			POST CURE	
	TIME, hr	TEMP., °C	PRESSURE, MPa	TIME, hr	TEMP., °C
POLYSTYRYL PYRIDINE (T-1, S-3)	4	200	1.01	NONE	
	2	250	1.01		
EPOXY (T-2, S-6, S-7, P-2)	3	177	0.69	NONE	
PHENOLIC — A(S-1)	4	130	0.69	4	121
PHENOLIC — B(S-2, S-4)	4	202	1.38	13	250
PHENOLIC — C(S-5)	4	149	0.69	NONE	
POLYIMIDE (P-1)	4	177	0.69	15	210

TABLE 3.- TEST RESULTS - BURN/IMPACT TEST

SAMPLE	RESIN TYPE	STYLE FIBER	RESIN CONTENT %/wt	% CHAR BINDER	% IMPACT LOSS
T-1	POLYSTYRYL PYRIDINE	UNIDIRECTIONAL TAPE	30.0	24.0	2.5
T-2	EPOXY	UNIDIRECTIONAL TAPE	30.0	13.1	12.6
S-1	PHENOLIC A	SATIN WEAVE	32.0	22.2	0.23
S-2	PHENOLIC B	SATIN WEAVE	30.0	20.7	0.40
S-3	POLYSTYRYL PYRIDINE	SATIN WEAVE	24.0	19.2	0.51
S-4	PHENOLIC B	SATIN WEAVE	21.5	18.1	0.62
S-5	PHENOLIC C	SATIN WEAVE	21.0	15.3	0.77
S-6	EPOXY	SATIN WEAVE	33.2	14.5	1.02
S-7	EPOXY	SATIN WEAVE	28.0	11.4	1.30
P-1	POLYIMIDE	PLAIN WEAVE	44.5	30.6	0.32
P-2	EPOXY	PLAIN WEAVE	28.0	11.4	0.90

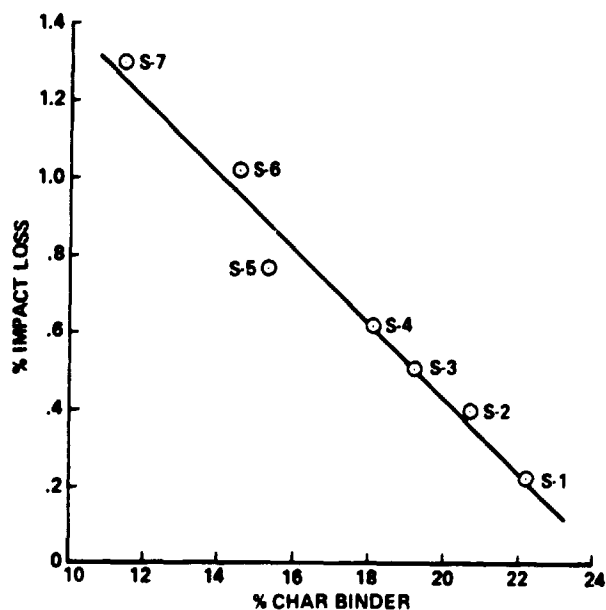


Figure 3.- Char vs impact loss after burn of satin weave composites.

CONCLUSIONS

The burn/impact chamber provides a small-scale test apparatus for determining a relative fiber-release value for burned carbon composites. The test results indicate a difference in carbon fiber release depending on the type of reinforcement used in the composite material. There was more fiber release with the quasi-isotropic composite made with unidirectional tape than with the woven fabric reinforcement. The amount of fiber release in the impact chamber after burning is coincident with the calculated char binder present.

One goal for our research effort is to reduce or eliminate the release of conductive fibers. The use of the burn/impact test chamber will assist in the evaluation of new resins and fibers.

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